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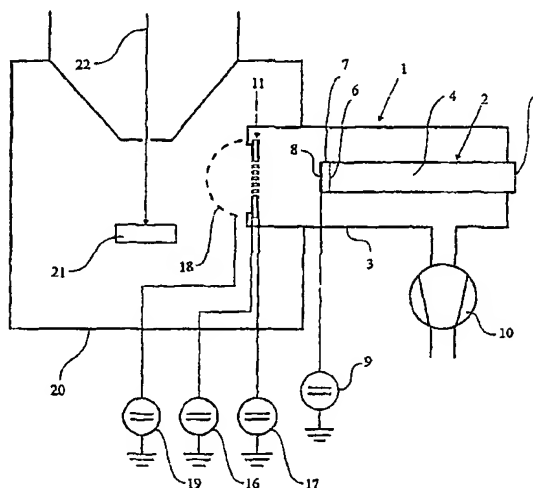
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(54) Title: **SECONDARY ELECTRON DETECTOR, ESPECIALLY IN A SCANNING ELECTRON MICROSCOPE**



(57) **Abstract:** The present invention deals with a secondary electron detector (1), especially in a scanning electron microscope. The subject matter of the invention provides a secondary electrons detector (1) constituted by a sensor (2) located in a detector chamber (3), to which a vacuum air pump (10) is connected to produce vacuum inside the detector chamber (3), the detector chamber (3) being in its wall near to the active surface of the sensor (2) enclosed with a diaphragm featuring high resistance to the transmission of gas and low resistance to the transmission of the electrons. The electrically conductive grid (11) is produced either in the form of a copper screen or as a capton membrane (12) with orifices (13) and it is equipped on both sides with conductive coating (14, 15). Outside the detector chamber (3), the electrically conductive grid (11) is covered with an input screen (18), which is usually of hemispherical shape and is connected to the low voltage source (19) of 80 to 150 V.

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SECONDARY ELECTRON DETECTOR, ESPECIALLY IN A SCANNING ELECTRON MICROSCOPE

Field of the invention

- 5 The invention relates to a secondary electron detector, especially in a scanning electron microscope.

Background of the invention

- 10 To create an image of a specimen by means of a scanning electron microscopy, a sample is scanned by an electron beam. Secondary electrons that are emitted from the sample by the striking electron beam are detected and by this signal a synchronously scanned electron beam in a monitor is modulated. The scanning usually takes place within a vacuum, especially to enable the action of both the electron gun and the electron detector.

- 15 The handling of a specimen within a vacuum presents a lot of problems. The biological specimens do not survive within a vacuum, wet specimens evaporate before an exact image can be produced. An observation of specimens from which different gaseous substances may leak within a vacuum of the microscope should be carefully considered in advance. Nonconductive samples within a vacuum
20 accumulate static charges on their surface, making thus precision microscopy practically impossible. This problem would be solved by coating such specimens, which, in some specimens such as semiconductors, may entail their destruction and thus it is practically impossible to carry out their nondestructive analysis.

- 25 A series of experiments has been carried out to observe specimens in which the vacuum of the microscope specimen chamber was separated from the source of the electron beam to enable the observation of the specimen while maintaining sufficient vacuum in the region of the electron gun. Patents US 4,785,182 and 4,880,976 by James F. Mancuso et al. describe a secondary electron detector to be used in gaseous atmosphere. In this case the vacuum of the electron gun and
30 electron microscope column is established to the value common in the electron microscopy. This part of the electron microscope is then separated from the specimen chamber by a pressure reducing diaphragm, which is substantially a lid enclosing this part of the electron microscope. A small orifice is made in the middle of the lid enabling the passage of the electron beam, nevertheless, it establishes

considerable resistance to the flow of gas from the specimen chamber to the electron gun. When high-efficiency vacuum pumps are used, the vacuum can be maintained at a value acceptable for the operation of the electron gun and in turn the gas pressure in the specimen chamber can be maintained at the level required by the specimen or sample. Between the sample and the pressure-limiting diaphragm a plate electrode with an orifice in the middle is located to enable the passage of the electron beam, to which electric potential is applied. The secondary electrons emitted from the sample are attracted by the electric field of the electrode and conveyed to the detector.

10 The evident disadvantage this arrangement suffers is the impossibility of simultaneously optimizing the pressure within the specimen chamber and the pressure in the detector chamber. If, in consideration of the biological samples and their survival within the vacuum, the pressure is maintained at a higher level, the conditions necessary for the operation of the detector are generally insufficient and, 15 vice versa, if the vacuum in the specimen chamber is established to an optimum value for the detector operation, the biological samples do not survive and no non-destructive observation can be performed.

WO 98/22971, applied for by Leo Electron Microscopy Limited, describes another system, in which negative voltage is applied to a sample holder and this 20 voltage repels secondary electrons emitted from the sample by the primary electron beam into a collision zone in a specimen chamber, in which the collisions of the accelerated secondary electrons and gas molecules in the gaseous medium triggers a cascade of collisions and thus generate an amplified signal of secondary electrons, which is detected in the microscope. This signal comprises photons generated by 25 the collisions of the accelerated secondary electrons and the gas molecules in the gaseous medium and it is detected by a photo-multiplier, to which the photons are conveyed via a light guide.

Nevertheless, this system has not solved thoroughly the problem of choking the secondary electrons signal by the back-scattered electrons signal. In addition to 30 it, the photon signal is usually very weak and the quality of the produced image is low.

UK Patent No. 2367686 by Leo Electron Microscopy Limited, describes another system that uses a detector chamber having a different vacuum value than the specimen chamber, both chambers being separated by a thin Al foil that should

prevent the gas from the specimen chamber to penetrate to the detector chamber while at the same time enable the electrons to pass through the foil from the specimen chamber to the detector chamber.

The disadvantage of this system is that it does not sufficiently differentiate
5 between secondary electron signal that gives a topographical information and a backscattered electrons signal that gives an information about the material of the specimen. Namely, a backscattered electron easily penetrates the Al foil and causes the emission of the secondary electrons from the surface of the foil that faces the scintillator. These electrons are then attracted by the scintillator and the signal with a
10 topographical information is thus mixed with a signal that bears an information about the material of the specimen. Further mixture of both types of information occur if the backscattered electron impinges, after passing through the Al foil, directly the scintillator. Another disadvantage is that the Al foil is too thin, about 7,5 nm, and is thus vulnerable to a damage in use. Further disadvantage is that the foil is in the
15 specimen chamber environment easily contaminated and considering how thin the foil is, the contamination layer acquires soon a thickness comparable with the thickness of the foil, what results in worse penetrability of low energy secondary electrons without influencing high energy backscattered electrons and thus in a worse efficiency of signal collection.

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Subject matter of the invention

The stated shortcomings of the previous state of the art are noticeably eliminated by a secondary electron detector, especially in a scanning electron
25 microscope, where the subject matter of the invention consists in the fact that the secondary electron detector is constituted by a sensor situated in a detector chamber, to which a vacuum pump is coupled to create a vacuum inside the detector chamber, while the detector chamber is closed in its wall near to an active surface of the sensor by a diaphragm featuring high resistance to a transmission of gas and low
30 resistance to a transmission of the electrons. All its remaining walls separate in a vacuum-tight manner the interior of the detector chamber from an ambient medium.

This diaphragm featuring high resistance to the transmittance of gas and low resistance to the transmittance of the electrons is constituted by an electrically conductive grid to which at least one bias voltage source is connected.

The electrically conductive grid is preferably made of copper or it is constituted by a diaphragm made of perforated electrical insulating material, for example capton, while this diaphragm is fitted with the first conducting coating on the side near to the sensor and with the second conducting coating applied to its reverse side, where the first conducting coating is electrically isolated from the second conducting coating.

The bias voltage source is preferably a source of bias voltage of 50 to 2000 V and even more preferably the source of bias voltage of 250 to 700 V.

In another embodiment of the secondary electron detector, the sensor is constituted by a light guide, with an ionization grid being arranged between its input and the electrically conductive grid and connected to the ionization voltage source, while the light guide output is led to the photo-multiplier input.

The preferred embodiment of the light guide is at its input fitted with a scintillator featuring a conducting coating on the side near to the electrically conductive grid, to which a high voltage source is connected.

In another exemplified embodiment, the sensor is constituted by a PIN diode.

In another exemplified embodiment, the sensor is fitted with a metallic disk to which a high voltage source and a current detector are connected.

In all the above described embodiments, the electrically conductive grid is outside the detector chamber preferably covered with an input screen, preferably of hemispherical shape, connected to a low voltage source of 50 to 500 V or preferably of 80 to 150 V.

Brief description of the drawings

The invention will be described in detail according to the enclosed drawings, where Fig. 1 shows the preferred embodiment of the secondary electron detector fitted with the capton diaphragm according to the present invention, in the specimen chamber of the electron microscope, Fig. 2 shows another preferred embodiment of the secondary electron detector with a copper grid, according to the present invention, also in the specimen chamber of the electron microscope, Fig. 3 shows the sectional view of the capton diaphragm used in the first exemplified embodiment of the detector according to this invention and Fig. 4 shows a preferred embodiment of the secondary electron detector with a metallic disk, according to the present invention, also in the specimen chamber of the electron microscope.

Description of preferred embodiments

Fig. 1 shows the first exemplified embodiment of the secondary electron detector according to the present invention. The secondary electrons detector 1 consists of the sensor 2 located in the chamber 3 of the detector. The sensor 2 consists of the light guide 4 to whose output 5 the photo-multiplier (not shown) is connected and to whose output 6 the scintillator 7 is connected, whose surface is coated, while to the coating 8 of the scintillator 7 the high voltage source 9 is connected. To the detector chamber 3 vacuum air pump 10 is connected to create vacuum inside the detector chamber 3. The wall of the detector chamber 3 near to the scintillator 7 is enclosed by an electrically conductive grid 11 constructed from diaphragm 12, in the given embodiment of capton, with orifices 13 and fitted on both sides with conducting coatings 14 and 15. The configuration of capton diaphragm 12 is shown in detail in Fig. 3. To both conducting coatings 14, 15 voltage sources 16, 17 are connected. To the first conducting coating 14 near to sensor 2 the source 16 of a voltage of 250 V is connected and to the second conducting coating 15 far from sensor 2 the source 17 of a voltage of 500 V is connected. The electrically conductive grid 11 is covered outside the detector chamber 3 with hemispherical input grid 18, which is connected to source 19 of low voltage of 80 to 150 V. Light guide 4 has its output 5 situated outside the detector chamber 3 and its passage through the wall of the detector chamber 3 is vacuum tight. The detector chamber 3 is located in the specimen chamber 20 of the electron microscope. Front panel 3 of the secondary electrons detector 1 fitted with hemispherical input grid 18 is near to the specimen 21 in the specimen chamber 20.

The secondary electrons detector 1 in the first exemplified embodiment works as follows: The electron beam 22 accelerated in the electron microscope strikes the surface of specimen 21 and the impinging electrons cause the emission of the secondary electrons from the surface of specimen 21, the energy of the emitted secondary electrons being only a few electronvolts, while the back-scattered electrons have the energy of several kiloelectronvolts. The back-scattered electrons are scattered to different directions and only a negligible number of them have the chance of passing through orifices 13 in capton diaphragm 12, as the electric field of the hemispherical input grid 18, to which a potential of 80 V is applied, has itself a negligible energy by comparison with the energy of the back-scattered electrons. By

contrast with this, the secondary electrons, in view of their low proper energy, are strongly attracted by the electric field of the hemispherical grid 18. Due to the gaseous atmosphere in the electron microscope specimen chamber 20, the electrons collide in course of their movement with the gas molecules and thus further

5 secondary electrons are created and thus the effect is multiplied. In the area under the hemispherical input grid 18 an electric field is produced by the 250 V voltage on the first conducting coating 14. This electric field creates electrostatic micro lenses at the holes in the hemispherical input grid 18 and it is transmitted through the holes outside the hemispherical input grid 18. The secondary electrons reaching the

10 surface of the hemispherical input grid 18 thus do not generally impinge on the hemispherical input grid 18, but pass through the holes in the grid to the compartment between the hemispherical input grid 18 and capton diaphragm 12. They are reattracted in this space by 250 V voltage on the capton diaphragm 12. Nevertheless, the second conducting coating 15 has a voltage of 500 V and thus in

15 orifices 13 analogous electrostatic lenses are created and the secondary electrons reaching the surface of capton diaphragm 12 mostly do not strike the first conducting surface 14 but pass through orifices 13 into the detector chamber 3. In this detector chamber 3, they are entrained by the electric field of the coating 8 of the scintillator 7. As it is known in the art, due to the voltage of 10 kV on the coating 8 of the scintillator

20 7, the secondary electrons pass through the coating 8 and strike the scintillator 7 with an energy of approximately 10 keV. The scintillations produced by the secondary electrons striking scintillator 7 are then transmitted through the light guide 4 to the photo-multiplier, on whose output a signal appears, whose value is in proportion to the number of secondary electrons emitted from the surface of the

25 specimen 21.

In experiments carried out with this arrangement, the capton diaphragm 12 was used. It was made of a capton foil 0.125 mm thick plated on both sides with aluminum and subsequently holes were drilled into it of a diameter of 0.15 mm in a square matrix of 10 x 10 holes with a spacing of 0.5 mm. The pressure in the

30 detector chamber 3 was established by the vacuum air pump 10 to a value of less than 7 Pa. With this arrangement, a satisfactory image was attained even with a primary beam current in the order of tens of pA.

Fig. 2 shows the second exemplified embodiment of the secondary electron detector according to the present invention. This embodiment differs from the first

exemplified embodiment by the fact that a copper grid was used in place of the electrically conductive grid 11. In this embodiment, the system of micro lenses on the electrically conductive grid 11 is omitted that was introduced by the different charges of the two coatings of the capton diaphragm 12, nevertheless, the electrically conductive grid 11 is located in a strong field of electric voltage of 10 kV on the coating 8 of the scintillator 7, so that a system of micro lenses is also created on the electrically conductive grid 11, which entrains the secondary electrons into the orifices 13 made in the electrically conductive grid 11 and further on to the scintillator 7. In view of the technological difficulties related to the production of the capton diaphragm 12 with the orifices 13, where the capton diaphragm 12 must be coated on both sides and the inner walls of the orifices 13 must retain their nonconductive surface, current capton diaphragms 12 compared to the current copper grids have a much smaller ratio of the area of orifices 13 to the area of the grid material. Thus, due to the current technology of manufacturing the capton diaphragms 12, the current copper grids achieve a signal by one order higher than the capton diaphragms 12.

Fig. 4 shows another exemplified embodiment of the secondary electron detector 1 according to the present invention. Sensor 2 is fitted with the metallic disk 23 to which the high voltage source 9 and the current detector 24 are connected.

The exemplified embodiment shown in Fig. 4 works as follows: both the secondary electrons, after having passed through conducting grid 11 and being accelerated by the high voltage from the source 9 and the electrons released in the course of ionization of the residual atmosphere inside the detector chamber 3 along the secondary electron path strike, instead of scintillator 7, the metallic disk 23 from where they are conveyed to the current detector 24. The signal detected by the current detector 24 corresponds to the signal of the secondary electrons emitted from the specimen surface.

Fig. 5 shows the fourth exemplified embodiment of the secondary electron detector 1 according to the present invention. This embodiment differs from the foregoing exemplified embodiments by the fact that the sensor 2 is designed as the light guide 4 between whose input 6 and the diaphragm 11 the ionization grid 25 connected to the ionization voltage source 26 is arranged. In this embodiment, in an analogous way to the foregoing embodiments, the secondary electrons are drawn into the detector chamber 3, where they reach the electric field of the ionization grid

25 by which they are attracted to the ionization grid 25. In this arrangement, the pressure in the detector chamber 3 is usually within the range of 20 to 100 Pa and the electrons are furthermore multiplied along the secondary electrons paths and thus the gas molecules are ionized in the detector chamber 3. In the course of this
5 ionization of the gas molecules, or during their transition to the basic condition, a fluorescence is produced and a light is emitted. A part of the produced light is conveyed via light guide 4 to the photo-multiplier (not shown).

In experiments with the arrangements according to the present invention, a standard copper grid used as a specimen holder in the transmission electron
10 microscopy was applied as electrically conductive grid 11. The active surface of the grid was approx. 2 mm in diameter and it contained approximately 160 orifices of 0.1 mm in diameter. Compared with the capton diaphragm 12 applied in the preceding embodiment, the area of the orifices was comparable, nevertheless, the orifices were concentrated on a ten times smaller area. A voltage of 400 to 500 V was applied to
15 the grid 11. In this arrangement, a signal approximately ten times stronger was attained than in the case in which the capton diaphragm 12 was applied and it appears to be the most effective state-of-the-art arrangement, with the added advantage of avoiding the technologically demanding production of the capton diaphragms 12.

20

Industrial applicability of the invention

The present invention may be utilized for the production of electron
microscopes, especially scanning electron microscopes, in which a higher gas
pressure in the microscope specimen chamber is required due to the character of the
25 examined specimen.

CLAIMS

1. A secondary electron detector, especially in a scanning electron microscope,
5 **characterized in that** it is comprised of a sensor (2) located in a detector chamber (3), to which a vacuum pump (10) is coupled to produce a vacuum inside the detector chamber (3), the detector chamber (3) being closed in the wall near to the active surface of the sensor (2) by a diaphragm featuring high resistance to a transmission of gas and a low resistance to a transmission of
10 electrons, while all its remaining walls vacuum-tightly separate the interior of the detector chamber (3) from the surrounding environment.
2. The secondary electron detector of claim 1, **characterized in that** the diaphragm featuring the high resistance to the transmission of gas and the low resistance to
15 the transmission of electrons is constituted by an electrically conductive grid (11) to which at least one source (16, 17) of bias voltage is connected.
3. The secondary electron detector of claim 2, **characterized in that** the electrically conductive grid (11) is made of copper.
20
4. The secondary electron detector of claim 2, **characterized in that** the electrically conductive grid (11) is constituted by a diaphragm (12) made of electrically insulating material provided with orifices (13), the diaphragm (12) being fitted with the first conductive coating (14) on the side near to the sensor (2) and with the
25 second conductive coating (15) applied to its reverse side, where the first conductive coating (14) is electrically insulated from the second conductive coating (15).
5. The secondary electron detector of claim 4, **characterized in that** the diaphragm
30 (12) is a capton diaphragm.
6. The secondary electron detector of any of the foregoing claims 2 to 5, **characterized in that** the source (16, 17) of bias is a source of bias of 50 to 2000 V.

7. The secondary electron detector of claim 6, **characterized in that** the source (16, 17) of bias voltage is a source of bias voltage of 250 to 700 V.
8. The secondary electron detector of any of the foregoing claims, **characterized in that** the sensor (2) consists of a light-guide (4), between whose input (6) and the electrically conductive grid (11) an ionization grid (25) is arranged that is connected to the source (26) of ionization voltage, while the light-guide output (4) leads to the photo-multiplier input.
9. The secondary electron detector of claim 8, **characterized in that** the light-guide (4) is at its input equipped with a scintillator (7), whose surface that is near to the electrically conductive grid (11) is fitted with a conductive coating (8), to which a high voltage source (9) is connected.
10. The secondary electron detector of any of the foregoing claims, **characterized in that** the sensor (2) is constituted by a PIN diode.
11. Secondary electron detector of any of the foregoing claims, **characterized in that** the sensor (2) is fitted with a metallic disk (23), to which a high a voltage source (9) and a current detector (24) are connected.
12. The secondary electron detector of any of the foregoing claims, **characterized in that** the electrically conductive grid (11) is covered outside the detector chamber (3) with an input screen (18), which is connected to a low voltage source (19) of 50 to 500 V.
13. The secondary electron detector of claim 12, **characterized in that** the electrically conductive grid (11) is outside the detector chamber (3) covered with an input screen (18), which is connected to the low voltage source (19) of 80 to 150 V.
14. The secondary electron detector of claim 12 or 13, **characterized in that** the input screen (18) is of hemispherical shape.

AMENDED CLAIMS

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Claim 1-14 replaced by claims 1-13.

CLAIMS

1. A secondary electron detector, especially in a scanning electron microscope,
5 **characterized in that** it is comprised of a sensor (2) located in a detector chamber (3), to which a vacuum pump (10) is coupled to produce a vacuum inside the detector chamber (3), the detector chamber (3) being closed in the wall near to the active surface of the sensor (2) by a diaphragm featuring high resistance to a transmission of gas and a low resistance to a transmission of electrons, while all its
10 remaining walls vacuum-tightly separate the interior of the detector chamber (3) from the surrounding environment, said diaphragm featuring the high resistance to the transmission of gas and the low resistance to the transmission of electrons being constituted by an electrically
15 conductive grid (11) to which at least one source (16, 17) of bias voltage is connected.
2. The secondary electron detector of claim 1, **characterized in that** the electrically conductive grid (11) is made of copper.
- 20 3. The secondary electron detector of claim 1, **characterized in that** the electrically conductive grid (11) is constituted by a diaphragm (12) made of electrically insulating material provided with orifices (13), the diaphragm (12) being fitted with the first conductive coating (14) on the side near to the sensor (2) and with the second conductive coating (15) applied to its reverse side, where the first conductive
25 coating (14) is electrically insulated from the second conductive coating (15).
4. The secondary electron detector of claim 3, **characterized in that** the diaphragm (12) is a capton diaphragm.
- 30 5. The secondary electron detector of any of the foregoing claims 1 to 4, **characterized in that** the source (16, 17) of bias is a source of bias of 50 to 2000 V.

6. The secondary electron detector of claim 5, **characterized in that** the source (16, 17) of bias voltage is a source of bias voltage of 250 to 700 V.
7. The secondary electron detector of any of the foregoing claims, **characterized in that** the sensor (2) consists of a light-guide (4), between whose input (6) and the electrically conductive grid (11) an ionization grid (25) is arranged that is connected to the source (26) of ionization voltage, while the light-guide output (4) leads to the photo-multiplier input.
8. The secondary electron detector of claim 7, **characterized in that** the light-guide (4) is at its input equipped with a scintillator (7), whose surface that is near to the electrically conductive grid (11) is fitted with a conductive coating (8), to which a high voltage source (9) is connected.
9. The secondary electron detector of any of the foregoing claims, **characterized in that** the sensor (2) is constituted by a PIN diode.
10. Secondary electron detector of any of the foregoing claims, **characterized in that** the sensor (2) is fitted with a metallic disk (23), to which a high a voltage source (9) and a current detector (24) are connected.
11. The secondary electron detector of any of the foregoing claims, **characterized in that** the electrically conductive grid (11) is covered outside the detector chamber (3) with an input screen (18), which is connected to a low voltage source (19) of 50 to 500 V.
12. The secondary electron detector of claim 11, **characterized in that** the electrically conductive grid (11) is outside the detector chamber (3) covered with an input screen (18), which is connected to the low voltage source (19) of 80 to 150 V.
13. The secondary electron detector of claim 11 or 12, **characterized in that** the input screen (18) is of hemispherical shape.

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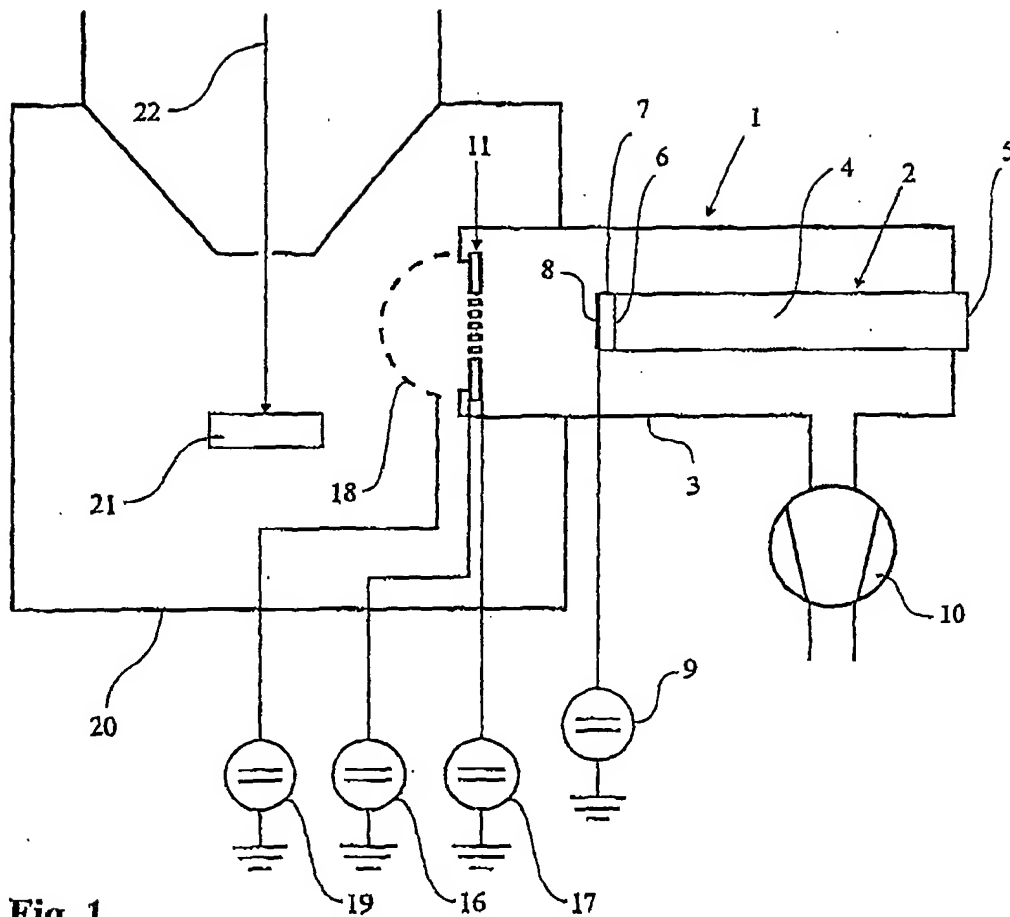


Fig. 1

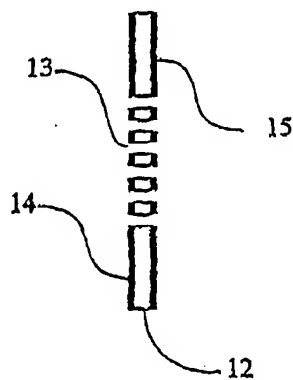


Fig. 3

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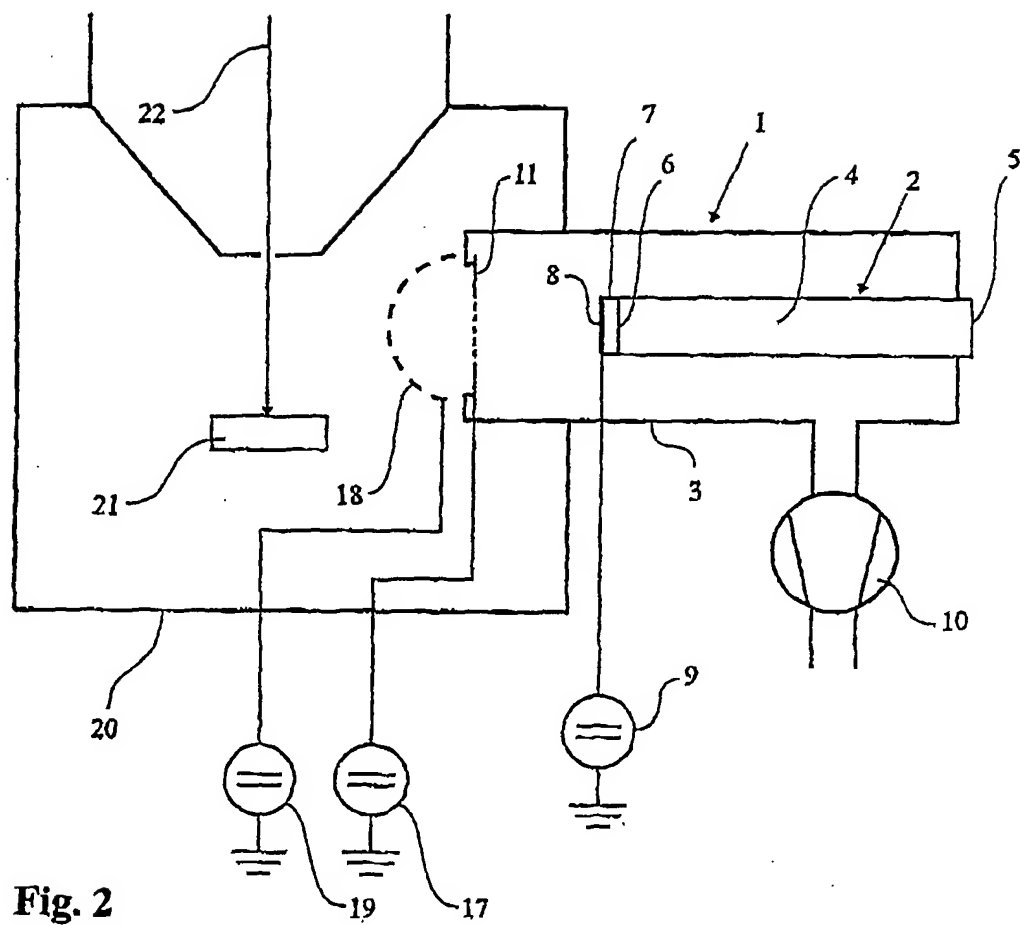


Fig. 2

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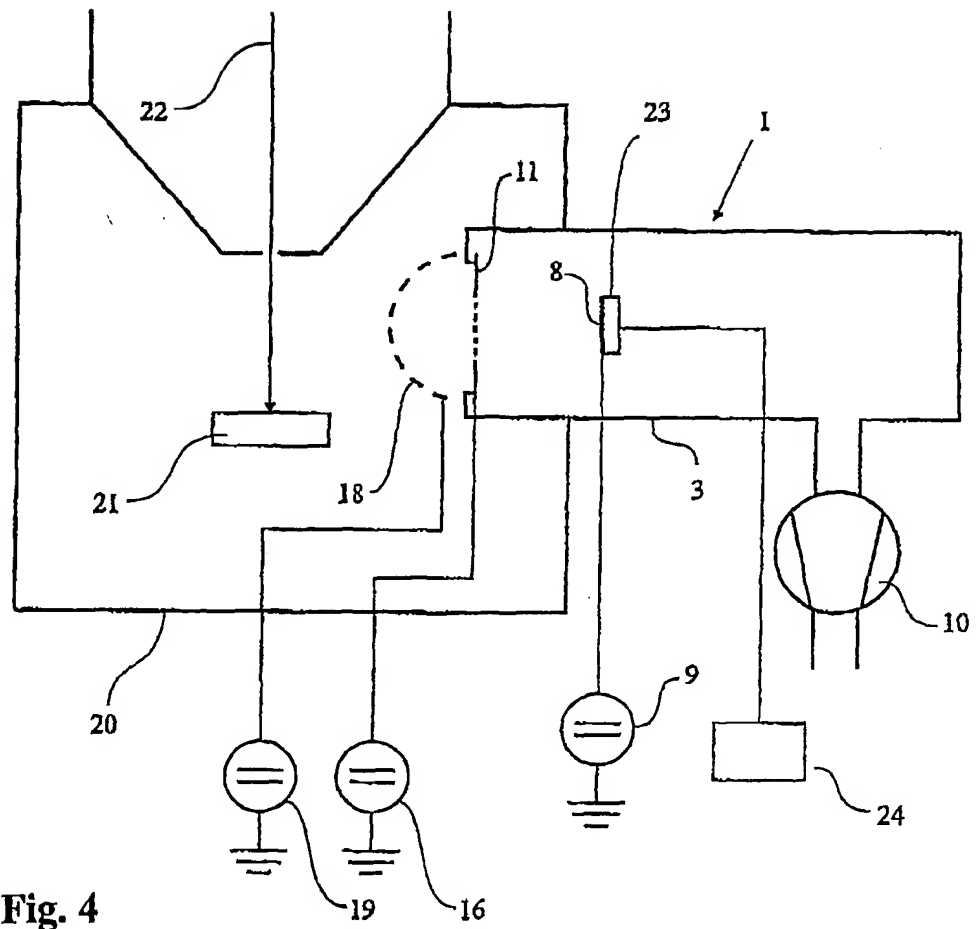
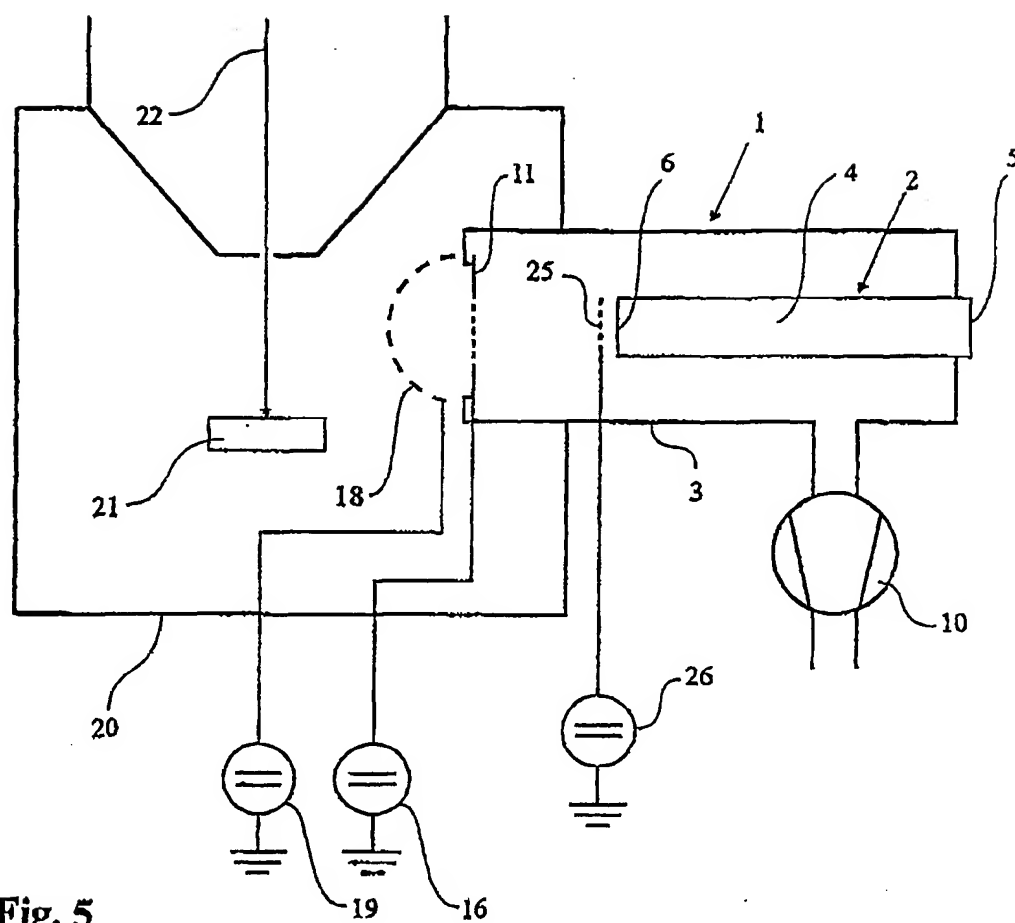


Fig. 4

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**Fig. 5**

INTERNATIONAL SEARCH REPORT

International Application No

PCT/CZ 03/00033

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01J37/244

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, PAJ, EPO-Internal, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>SLOWKO W: "Secondary electron detector with a micro-porous plate for environmental SEM"</p> <p>INTERNATIONAL SYMPOSIUM 'ION IMPLANTATION AND OTHER APPLICATIONS OF IONS AND ELECTRONS - ION 2000', KAZIMIERZ DOLNY O/WISLA, POLAND, 12-15 JUNE 2000, vol. 63, no. 4, pages 457-461, XP002268265</p> <p>Vacuum, 16 Aug. 2001, Elsevier, UK</p> <p>ISSN: 0042-207X</p> <p>page 459, left-hand column, paragraph 3</p> <p>-page 460, right-hand column, paragraph 2; figures</p> <p style="text-align: center;">--- -/-</p>	1

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

27 January 2004

Date of mailing of the international search report

13/02/2004

Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/CZ 03/00033

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>EP 1 179 833 A (LEO ELECTRON MICROSCOPY LTD) 13 February 2002 (2002-02-13) cited in the application paragraph '0028! - paragraph '0039!; figures</p> <p>-----</p>	<p>1,2,6-9, 12-14</p>

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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